

# (12) United States Patent

### Tanaka et al.

### (54) ADAMANTANE DERIVATIVE AND PROCESS FOR PRODUCING THE SAME

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U.S. Cl. ..... 568/665

Field of Classification Search ...... 568/665 See application file for complete search history.

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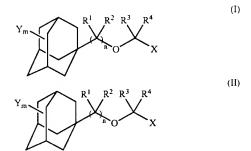
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Primary Examiner—Rei-tsang Shiao Assistant Examiner—Joseph R Kosack (74) Attorney, Agent, or Firm-Oblon, Spivak, McClelland, Maier & Neustadt, P.C.

#### **ABSTRACT** (57)

Provided is an adamantane derivative represented by Formula (I) or (II):



wherein X represents a halogen atom; Y represents an alkyl group having 1 to 10 carbon atoms, a halogenated alkyl group having 1 to 10 carbon atoms, a halogen atom or a hetero atom-containing group; R1 to R4 represent independently hydrogen, a halogen atom, an alkyl group having 1 to 10 carbon atoms or a halogenated alkyl group having 1 to 10 carbon atoms; m represents an integer of 0 to 15, and n represents an integer of 0 to 10; and excluded is a case where in Formula (I), m and n are 0 at the same time and R3 and R4 are a hydrogen atom at the same time.

Capable of being provided is a novel adamantane derivative which is useful as a modifying agent for a resin for a photoresist and a dry etching resistance-improving agent in the photolithography field, agricultural and medical intermediates and other various industrial products.

#### 3 Claims, No Drawings

A separable flask having a volume of one liter equipped with a nozzle for introducing hydrogen chloride gas was equipped with a stirring device and charged with 50.0 g (301 mmol) of 4-oxo-2-adamantanol, 13.6 g (450 mmol) of paraformaldehyde, 36.2 g (300 mmol) of magnesium sulfate and 650 ml of dried dichloromethane, and it was cooled to 0° C. on an ice bath and stirred. Hydrogen chloride gas generated by mixing 300.7 g (5 mole) of sodium chloride with 700 ml of 25 conc. sulfuric acid was blown thereinto through the nozzle for one hour. After further stirring for 3 hours, magnesium sulfate was filtered, and then the solution was analyzed by gas chromatography to confirm that 4-oxo-2-adamantanol was completely converted and that the intended product was obtained at a selectivity of 93.2%. Hydrogen chloride and dichloromethane were removed, and then refining was carried out by distillation to isolate 55.0 g (256 mmol, yield 85.2%, GC purity 98.8%) of the intended product.

The analytical results of the above compound are shown below

Nuclear magnetic resonance spectrometry (NMR): CDCl<sub>3</sub>

<sup>1</sup>H-NMR (500 MHz): 1.66 to 1.69 (m), 1.75 to 1.78 (m),
1.89 to 2.12 (m), 2.20 (m), 2.25 (m), 2.82 (m), 2.40 (dq, 40 J=13.0 Hz, 2.8 Hz), 2.51 (s), 2.54 (s), 2.79 (s), 3.94 (t, J=3.5 Hz, 1H, b²), 4.31 (q, J=2.7 Hz, 1H, b¹), 5.52 (s, 2H, a¹), 5.55 (dd, J=5.4 Hz, 17.6 Hz, 2H, a²), <sup>13</sup>C-NMR (127 MHz): 26.26 (g¹), 26.39 (g²) 29.98 (f² or h² or j² or k²), 30.95 (i²), 31.26 (i¹), 32.46 (f² or h² or j² or k²), 32.98 (f¹ or h¹ or j¹ or k¹), 33.44 (f² or h² or j² or k²), 34.99 (f¹ or h¹ or j¹ or k¹), 37.80 (f¹ or h¹ or j¹ or k¹), 38.68 (f² or h² or j² or k²), 38.78 (f¹ or h¹ or j¹ or k¹), 45.31 (e²), 46.18 (e¹), 50.52 (c¹), 51.07 (c²), 79.69 (b¹), 79.82 (a²), 80.44 (b²), 83.97 (a¹), 213.86 (d²) 214.96 (d¹) (superscript numeral 1 represents a principal isomer, and superscript numeral 2 represents a subsidiary isomer)

Gas chromatography mass spectrometry (GC-MS): EI 216 (M<sup>+</sup>+2, 2.9%), 214 (M<sup>+</sup>, 8.7%), 148 (29.2%), 79 (100%) 55 Boiling point: 160 to 161° C./0.2 kPa

### INDUSTRIAL APPLICABILITY

The adamantane derivatives of the present invention are novel (monohalogen-substituted methyl) (adamantyl group-containing alkyl)ethers, and they are useful as a modifying agent for a resin for a photoresist and a dry etching resistance-improving agent in the photolithography field, agricultural and medical intermediates and other various industrial products.

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What is claimed is:

1. A substituted adamantane of Formula (I) or (II):

$$Y_{m}$$

$$R^{1}$$

$$R^{2}$$

$$R^{3}$$

$$X$$

$$Y_{m}$$

$$R^{1}$$

$$R^{2}$$

$$R^{3}$$

$$R^{4}$$

$$R^{1}$$

$$R^{2}$$

$$R^{3}$$

$$R^{4}$$

$$R^{4}$$

$$R^{1}$$

$$R^{2}$$

$$R^{3}$$

$$R^{4}$$

$$R^{4}$$

wherein

X represents a halogen atom;

Y is a C<sub>1</sub>-C<sub>10</sub> alkyl group, a halogenated C<sub>1</sub>-C<sub>10</sub> alkyl group, a halogen atom or a hetero atom-containing group;

 $R^1$  and  $R^2$  represents, independently, hydrogen, a halogen atom, a  $C_1$ - $C_{10}$  alkyl group or a halogenated  $C_1$ - $C_{10}$  alkyl group;

 $R^3$  and  $R^4$  represent, independently, hydrogen, a  $C_1$ - $C_{10}$  alkyl group, or a  $C_1$ - $C_{10}$  halogenated alkyl group,

wherein R<sup>3</sup> and R<sup>4</sup> cannot both be hydrogen in Formula (I); wherein, in the formula (I), m represents an integer of 0 to 15, and n represents an integer of 1 to 10;

wherein, in the Formula (II), m represents an integer of 1 to

2. A substituted adamantane selected from the group consisting of

$$Ym$$
 $O$ 
 $R^3$ 
 $R^4$ 
 $X$ 

$$\begin{array}{c} \text{Ym} \\ \text{O} \\ \text{O} \\ \text{X}, \end{array}$$

$$\begin{array}{c} \text{Ym} \\ \text{O} \\ \text{O} \\ \text{X}, \end{array} \tag{IIe}$$

$$\begin{array}{c} Ym \\ O \\ \end{array} \begin{array}{c} R^3 \\ X, \end{array} \tag{IId}$$

(Ia)

10

20

(Ic) 25

(Id) 30

40

45

(If)

-continued

$$R^1$$
  $R^2$   $R^3$   $R^4$  and

wherein

X represents a halogen atom;

(IIe) Y is a C<sub>1</sub>-C<sub>10</sub> alkyl group, a halogenated C<sub>1</sub>-C<sub>10</sub> alkyl group, a halogen atom or a hetero atom-containing group;

R1 and R2 represent, independently, hydrogen, a halogen atom, a C<sub>1</sub>-C<sub>10</sub> alkyl group or a halogenated C<sub>1</sub>-C<sub>10</sub> alkyl group;

R<sup>3</sup> and R<sup>4</sup> represent, independently, hydrogen, a C<sub>1</sub>-C<sub>10</sub>

alkyl group, or a  $C_1$ - $C_{10}$  halogenated alkyl group, wherein  $R^3$  and  $R^4$  cannot both be hydrogen in Formulae (Ia-If);

wherein, in the formulae (Ia-If), m represents an integer of 0 to 13, and n represents an integer of 1 to 10;

wherein, in the Formula (IIa-IIe), m represents an integer 15 of 1 to 13.

(Ib) 3. A process for producing the substituted adamantane of claim 1, comprising

reacting an alcohol comprising an adamantyl group represented by Formula (III) or (IV):

$$Y_m$$
 (III)

$$Y_m$$
 $R^1$ 
 $R^2$ 
 $OH$ 

wherein in Formula (III), n represents an integer of 1 to 10; 35 with a carbonyl compound represented by Formula (V): (le)

$$R_3$$
  $\longrightarrow$   $R_4$   $(V)$ 

and at least one hydrogen halide gas; wherein R3 and R4 in Formula (V) are not both hydrogen when the carbonyl compound of formula (V) is reacted with the adamantyl group of Formula (III), and wherein m represents an integer of 1 to 13.

DOCKET NO: 273504US0PCT

# IN THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE APPLICATION OF :

SHINJI TANAKA, ET AL. : EXAMINER: J. R. KOSACK

SERIAL NO: 10/540,547 :

FILED: DECEMBER 13, 2005 : GROUP ART UNIT: 1626

FOR: ADAMANTANE DERIVATIVE AND PROCESS FOR PRODUCING THE

SAME

# AMENDMENT AND REQUEST FOR RECONSIDERATION

COMMISSIONER FOR PATENTS ALEXANDRIA, VIRGINIA 22313

SIR:

In response to the Office Action dated March 19, 2008, please amend the aboveidentified application as follows:

Amendments to the Claims are reflected in the listing of claims which begins on page 2 of this paper.

Remarks/Arguments begin on page 6 of this paper.

# IN THE CLAIMS

Please amend the claims as follows:

Claim 1 (Currently Amended): [[An]] A substituted adamantane derivative characterized by having a structure represented by of Formula (I) or (II):

$$Y_{n}^{m} \xrightarrow{R^{1} R^{2} R^{3} R^{4}} X \dots (I)$$

wherein

X represents a halogen atom;

Y represents an is a  $C_1$ - $C_{10}$  alkyl group having 1 to 10 carbon atoms, a halogenated  $C_1$ - $C_{10}$  alkyl group having 1 to 10 carbon atoms, a halogen atom or a hetero atom-containing group;

 $R^1$  [[to  $R^4$ ]] and  $R^2$  represent, independently, hydrogen, a halogen atom, [[an]] a  $C_{1-}$   $C_{10}$  alkyl group having 1 to 10 carbon atoms or a halogenated  $C_{1-}C_{10}$  alkyl group having 1 to 10 carbon atoms;

 $R^3$  and  $R^4$  represent, independently, hydrogen, a  $C_1$ - $C_{10}$  alkyl group, or a  $C_1$ - $C_{10}$  halogenated alkyl group,

wherein R<sup>3</sup> and R<sup>4</sup> cannot both be hydrogen in Formula (I);
wherein, in the formula (I), m represents an integer of 0 to 15, and
n represents an integer of 1 to 10 0 to 10; and

wherein, in the Formula (II), m represents an integer of 1 to 15

excluded is a case where in Formula (I), m and n are 0 at the same time and R<sup>3</sup> and R<sup>4</sup> are a hydrogen atom at the same time.

Claim 2 (Currently Amended): [[The]] A substituted adamantane selected from the group consisting of

# wherein

X represents a halogen atom;

Y is a  $C_1$ - $C_{10}$  alkyl group, a halogenated  $C_1$ - $C_{10}$  alkyl group, a halogen atom or a hetero atom-containing group;

 $R^1$  and  $R^2$  represent, independently, hydrogen, a halogen atom, a  $C_1$ - $C_{10}$  alkyl group or a halogenated  $C_1$ - $C_{10}$  alkyl group;

 $R^3$  and  $R^4$  represent, independently, hydrogen, a  $C_1$ - $C_{10}$  alkyl group, or a  $C_1$ - $C_{10}$  halogenated alkyl group,

wherein R<sup>3</sup> and R<sup>4</sup> cannot both be hydrogen in Formulae (Ia-If);
wherein, in the formulae (Ia-If), m represents an integer of 0 to 13, and
n represents an integer of 1 to 10 0 to 10; and

wherein, in the Formula (IIa-IIe), m represents an integer of 1 to 13

derivative according to claim 1, wherein in Formula (I) or (II), Y represents =0

formed by allowing two Y's to be put together.

Claim 3 (Currently Amended): A process for producing the <u>substituted</u> adamantane derivative of claim 1, comprising

reacting an alcohol comprising an adamantyl group represented by Formula (III) or (IV):

wherein in Formula (III), n represents an integer of 1 to 10;

wherein  $X, Y, R^1, R^2$ , m and n are the same as described above, with a carbonyl compound represented by Formula (V):

and at least one hydrogen halide gas; to obtain the adamantine derivative: wherein R<sup>3</sup> and R<sup>4</sup> are the same as described above, and wherein when m and n are 0 at the same time in Formula (III) described above, R<sup>3</sup> and R<sup>4</sup> in Formula (V) are not both hydrogen when the carbonyl compound of formula (V) is reacted with the adamantyl group of Formula (III), and wherein m represents an integer of 1 to 13 are not a hydrogen atom.

Claims 4-8 (Cancelled).

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## REMARKS/ARGUMENTS

Claims 4-8 are cancelled.

Support for each amended claim is found at the originally filed claims and throughout the specification.

Upon entry of the amendment, Claims 1-3 will be active.

No new matter is believed to have been added.

Any duplicate claim objection of Claim 8 is obviated by cancellation of Claim 8.

The enablement rejection of Claims 3-8 is respectfully traversed. The rejection of Claims 4-8 is obviated by cancellation of these claims. Present Claim 1, and by extension present dependent Claim 3, has been amended to contain the features "R³ and R⁴ represent, independently, hydrogen, a C<sub>1</sub>-C<sub>10</sub> alkyl group, or a C<sub>1</sub>-C<sub>10</sub> alkyl group..." Because Claims 1 and 3 do not describe that R³ and / or R⁴ can be a halogen, Claims 1 and 3 are believed to be definite. Withdrawal of enablement rejection is respectfully requested.

The indefiniteness rejection of Claims 2, 5 and 7 is respectfully traversed. The rejection of Claims 5 and 7 is obviated by cancellation of these claims. Claim 2 has been amended such that the term "by allowing two Y's to be put together" has been removed from Claim 2. Accordingly, it is believed that Claim 2 is definite. Withdrawal of the rejection is respectfully requested.

The indefiniteness rejection of Claims 1-8 for containing features such as "having 1 to 10 carbon atoms" is respectfully traversed. The rejection of Claims 4-8 is obviated by cancellation of these claims. Applicants respectfully submit that one of ordinary skill in the art would readily understand what the phrase "having 1 to 10 carbon atoms" means, and therefore that the term is definite. Nevertheless, as a courtesy to the Office, Applicants have

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Reply to Office Action of March 19, 2008

replaced the phrase "having 1 to 10 carbon atoms" with the phrase " $C_1$ - $C_{10}$ ." Claims 1-3 are

definite. Withdrawal of the rejection is requested.

The anticipation rejection of Claims 1-8 as being unpatentable in view of Ben-David

is respectfully traversed. The rejection of Claims 4-8 is obviated by cancellation of these

claims. Present Claim 2 comprises oxoadamantyl moieties. These moieties are not

described or suggested by Ben-David. Further, present Claims 1 and 3 do not encompass the

compound of Ben-David cited at page 6 of the Official Action. Ben-David therefore cannot

anticipate present Claims 1-3. Withdrawal of the anticipation rejection is requested.

The obviousness rejection of Claims 4, 6 and 8 as being unpatentable in view of

Okada is obviated by cancellation of these claims.

Applicants submit the present application is now in condition for allowance. Early

notification to this effect is earnestly solicited.

Respectfully submitted,

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22850

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(OSMMN 08/07)

Charles J. Andres, Jr., Ph.D.

Attorney of Record

Registration No. 57,537

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irst Named Inventor:	Shinji Tanaka , Chiba, (JP)	Issue Date of Patent:	12-30-2008	

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0/540,547 ADAMANTANE DERIVATIVE AND PROCESS FOR PRODUCING THE SAME

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1-14-2009	COCIN	Request for Certificate of Correction	PROSECUTION	4				
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3-03-2008	IIFW	Issue Information including classification, examiner, name, claim, renumbering, etc.	PROSECUTION	1				
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5-27-2008	CLM 7	Claims	PROSECUTION	4				
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3-24-2005	SPEC	Specification	PROSECUTION	25
3-24-2005	CLM	Claims	PROSECUTION	3
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3-24-2005	A.PE	Preliminary Amendment	PROSECUTION	1
3-24-2005	CLM	Claims	PROSECUTION	7
3-24-2005	REM	Applicant Arguments/Remarks Made in an Amendment	PROSECUTION	1
3-24-2005	ADS	Application Data Sheet	PROSECUTION	3
3-24-2005	IDS	Information Disclosure Statement (IDS) Filed (SB/08)	PROSECUTION	2
3-24-2005	NPL	NPL Documents	PRIOR ART	5
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3-24-2005	NPL	NPL Documents	PRIOR ART	7
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3-24-2005	IMIS	Miscellaneous Internal Document	PROSECUTION	1
3-24-2005	371P	Documents submitted with 371 Applications	PROSECUTION	1
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